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## INSTRUMENTATION FOR SURFACE STUDIES FOR NOVEL ELECTRONICS PROCESSING

Columbia Microelectronics Sciences Laboratories

and

Columbia Radiation Laboratory

Final Technical Report

for

Air Force Office of Scientific Research

December 15, 1984 - December 14, 1985

R.M. Osgood

Principal Investigator

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(8) Principal Investigator: Richard M. Osgood, Jr.

Telephone Number: (212) 280-4462

(9) Program Manager: Dr. Howard Schlossberg

Telephone Number: (202) 767-4906

(10) Title: Instrumentation for surface studies for Novel

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The views and conclusions contained in this document are those of the authors and should not be interpreted as necessarily representing the official policies, either expressed or implied, of the Defense Advanced Research Projects Agency or the U.S. Government.

#### DESCRIPTION OF INSTRUMENTATION PURCHASED

#### I. <u>INTRODUCTION</u>

The instrumentation purchased on this contract is to be used in the study of solid-surface phenomena and the relation of these phenomena to developing new processing or fabrication techniques for micro-

electronics. This work represents the combined research programs of the Columbia Microelectronics Sciences Laboratory including Professors R.M. Osgood, E. Yang, and E.R. Fossum, and two professional staff members, Drs. H.H. Gilgen and Dr. Chienfan Yu.

The instrumentation purchased has already been used to prepare and diagnose sample surfaces for studies of microelectronics-processing-related phenomena, with particular emphasis on laser-surface and ion-surface chemical interactions. Development of new processing techniques including laser direct writing, photon-assisted dry processing, the formation of metal silicides, and ion etching are currently major research efforts in the Columbia Microelectronics Sciences Laboratories. The work is supported by research contracts with AFOSR, DARPA, JSEP, and Rome Air Development Center. This contract was for the basic instrumentation to support this surface oriented research including an ultrahigh-vacuum system, with diagnostics, and a chamber with mass spectrometry

#### II. DEMONSTRATED USE

The equipment purchased with this contract has already been used for several key experiments within the Columbia Microelectronics Sciences Laboratories. These experiments and the equipment itself is described in the following sections.

A. Surface Analysis System -- The UHV surface analysis system, made by Leybold Heraeus, was received in September, 1985. Within a few weeks, it had already been used to analyze the surface chemistry observed in ultraviolet laser-induced oxidation of GaAs. A very important partner in setting up this research apparatus was the IBM Corporation which, through the auspices of Dr. Daniel Grischowsky, a former Radiation Laboratory student, contributed a Post-Doctoral Fellowship for the person involved in the research on the apparatus.

The UHV machine is the first surface analysis system for solid-state research at Columbia. It is equipped for ESCA, Auger, and SSI analysis. In addition, it comes with a load-lock system for easy sample insertion.

This surface analysis system was first used to characterize a gallium arsenide surface oxidized by ultraviolet light in deionized water. In Fig. 1, X-ray photoelectron spectroscopy (XPS) reveals the change of chemical bonding on the gallium arsenide surface after etching. The decrease of the kinetic energy of the  $2P_{3/2}$  and 3d peaks (about 3 eV for arsenic and 1 eV for gallium) indicates the formation of  $Ga_2O_3$  and  $As_2O_3$  during the oxidation process. The drastic decrease of arsenic peak intensity also shows the effective removal of arsenic oxide by water.

Auger electron spectra (AES), shown in Fig. 2, were used to monitor the evolution of surface composition as the etched gallium arsenide surface was sputtered with argon ions. The oxide concentration is about 40% on the surface (normalized to the relative sensitivity) and decreases as a function of depth (sputtering time). On the other hand, arsenic concentration is less than 10% on the

surface and increses as probing goes deeper into the surface. The GaAs stoichiometry is finally restored at the depth of c.a. 500  $\text{\AA}$ .

Incorporation of this surface analysis system with a laser-surface etching system currently under construction has been conceived. Once realized, surface characterization and/or diagnosis can be made right after the surface is prepared and transferred to the analysis chamber without breaking the vacuum.

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Work is also being done on analyzing ion-beam-assisted oxidation of silicon - a project directed by Professor Fossum.

B. <u>Use of the Time of Flight Chamber for Analysis of the</u>

<u>Fundamental Chemistry of Laser Induced Reacitons</u> -- We have contructed a high-vacuum to UHV time-of-flight measurement chamber with time resolution of 10 nsec. The system also has an auxiliary unit that can carry out high pressure (1 μ- torr to 100 torr) etching processes. A schematic is shown in Fig. 3. The purpose of this system is to determine the GaAs/HBr etching mechanism, the energy distribution functions of the etch product desorbing from the surface adlayer in the GaAs/HBr system, and to determine the cause of crystallographic etching in this system.

Time-resolved laser-induce surface mass spectroscopy requires high time resolution and a good signal-to-noise ratio. We have purchased an Extra Nuclear C-50 quadrupole mass spectrometer with 10 nsec response time. The time-resolved mass signal is then digitized with a LeCroy built CAMAC system, averaged and recorded on an IBM/AT. The LeCroy TR8818/MM8103A transient digitizer can handle signals up to 100 MHZ and has 32K x 8 bit buffer memory. Digitized signal averaging and recording is done in the IBM/AT computer. We

have also purchased a discriminator, LeCroy 821, and multichannel, Scalar 352 a 100 MHZ, as a multi-channel analyzer for the desorbed species emission and mass spectrum. The emission detection can together give a positive species identification. So far, the computer - CAMAC interface has been tested and the CAMAC is ready to receive signal from the C-50 mass spectrometer.

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The vacuum system (Fig. 1) consists of the main chamber, mass spectrometer chamber, molecular beam chamber and auxiliary chamber. The main chamber is where the laser-surface interaction takes place. The mass spectrometer chamber is backed by a cryopump, and the mass spectrometer ionizer head is cooled and further pumped by an apertured liquid-nitrogen shroud to decrease the noise. The pulsed, molecularbeam source doses the sample surface with high density and monoenergetic parent gases. Radicals can be generated right at the surface when synchronized with a photodissociating laser pulse. beam machine is designed to produce a beam waist of 5 mm at the sample surface. A Newport Research electromagnetic valve driver is used to produce a nozzle-emission duration as short as 100-usec pulse. auxiliary pumping system (i) roughs all systems from atmosphere to 5 mtorr, (ii) roughs the mass spectrometer and cryopump to below 10<sup>-6</sup> torr, (iii) acts as a high vacuum back-up to all systems in the event of emergency and cold trap pumping, (iv) serves to allow low vacuum to UHV pumping for the fabrication process chamber which is an entirely LF flanged constructed cell for etching and deposition with the presence of ambient gas, and (v) provides main-chamber-sample load-lock vacuum and high vacuum pumping. So far, the main chamber, mass spectrometer chamber, and the molecular chamber are being

assembled in our lab.

In the next month time period, all vacuum system are expected to be leak checked; the fabrication process chamber is then expected to be completed. All electronics including the CAMAC crate and computer are expected to be lab mounted. After pump down is achieved, the molecular beam will be calibrated for mean speed and velocity profile. During this time, the CAMAC/PC wave form processor will be run with a simulation input signal.

In two months time, radicals are expected to be generated at the sample surface and the C-50 is expected to analyze simple, vacuum-ablation, surface-desorption products for calibration.

### III. MATCHING FUNDS

The instrumentation described above was assisted by three forms of matching funds:

- A) The University contributed approximately \$30,000 of matching funds to buy computer and signal processing equipment for the time of flight mass spectrometer. This equipment enables signal averaged detection of very low-concentration species.
- B) Part of the instrumentation was placed in a laboratory room refurbished for the equipment by the university.
- C) The IBM Corporation, through the Columbia Radiation
  Laboratory, contributed an IBM postdoctoral fellowship for a
  professional, Dr. Chienfan Yu, to work on bringing the instrumentation
  on line.

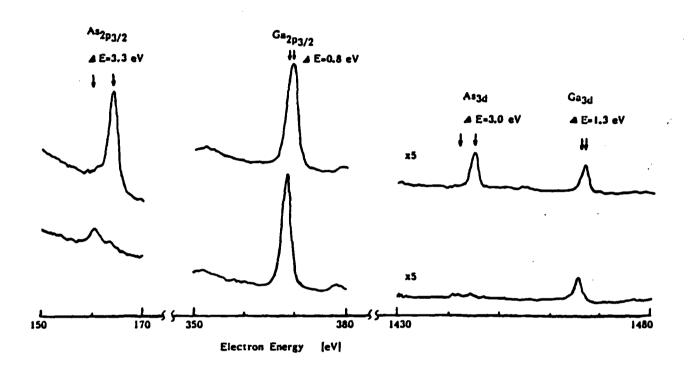


Figure 1: Photoelectron spectra of virgin (upper) and etched (lower) gallium arsenide surfaces. The aluminum K--line was used as the exciation source.

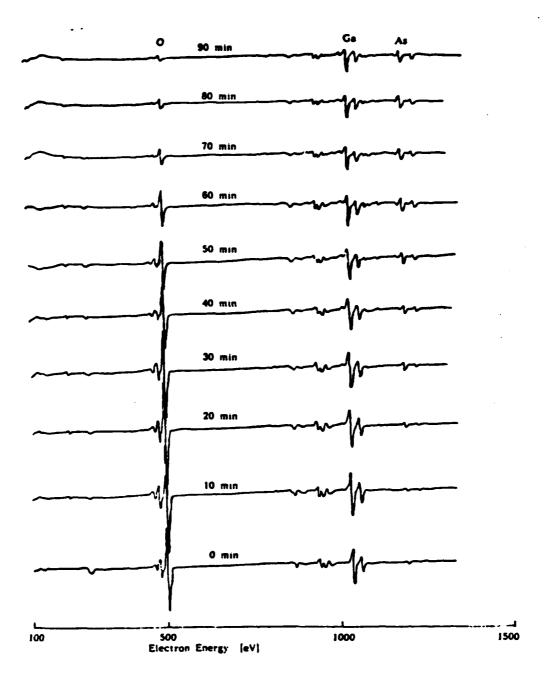


Figure 2: Evolution of the Auger electron spectra of etched gallium arsenide surface when sputtered with 3KV argon ion of 1.5  $\mu A/cm^2$  at 10 min. intervals.

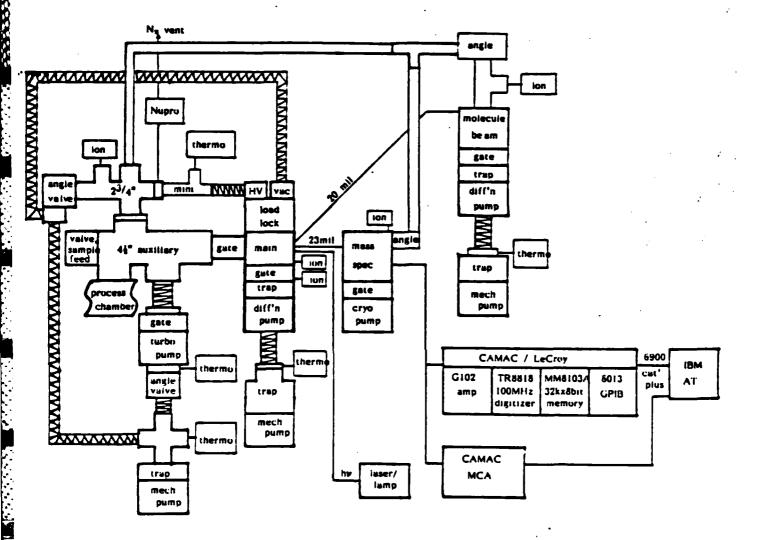


Figure 3: Schematic of time-of-flight measuring system.

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